

$\Delta/\sigma_{\max} < 0.01$ , maximum and minimum heights in final  $\Delta F$  map  $+0.17$  and  $-0.12 \text{ e } \text{\AA}^{-3}$ . The atomic scattering factors were from *International Tables for X-ray Crystallography* (1974). Computations performed with *NRCVAX* (Gabe, Lee & Le Page, 1985). The final positional and thermal parameters for the atoms are given in Table 1 while bond distances and angles, and torsional angles are listed in Table 2.\* The numbering scheme used is shown in the *ORTEPII* (Johnson, 1976) drawing of Fig. 1.

**Related literature.** In their previous report (Hughes & Nowell, 1978), the authors discussed torsional angles about the  $C_r-O$  bonds to show the strain in dibenzo-12-crown-4 ether. Related structural studies include dicyclohexyl derivatives of the 14-crown-4 ether (Buch-

anan, Kirby & Charland, 1988*b*), 12-crown-4 ether (Buchanan, Kirby & Charland, 1988*a*) and 13-crown-4 ether families (Buchanan, Kirby & Charland, 1988*c*).

#### References

- BUCHANAN, G. W., KIRBY, R. A. & CHARLAND, J.-P. (1988*a*). *J. Am. Chem. Soc.* Submitted.  
 BUCHANAN, G. W., KIRBY, R. A. & CHARLAND, J.-P. (1988*b*). *J. Am. Chem. Soc.* **110**, 2477–2483.  
 BUCHANAN, G. W., KIRBY, R. A. & CHARLAND, J.-P. (1988*c*). In preparation.  
 GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *Crystallographic Computing 3*, edited by G. SHELDRICK, C. KRUGER & R. GODDARD, pp. 167–174. Oxford: Clarendon Press.  
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.  
 HUGHES, D. L. & NOWELL, I. W. (1978). *J. Chem. Res. (M)*, pp. 1534–1556.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)  
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 LE PAGE, Y., GABE, E. J. & WHITE, P. S. (1987). *Acta Cryst.* **A43**, C-271.

\* Lists of observed and calculated structure factors and anisotropic temperature factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51295 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

*Acta Cryst.* (1989). **C45**, 167–168

## Structure of a Hetero-Diels–Alder Adduct

BY JOCHEN ANTEL AND GEORGE M. SHELDRICK

*Institut für Anorganische Chemie der Universität, Tammannstraße 4, D-3400 Göttingen, Federal Republic of Germany*

AND THOMAS BRUMBY AND LUTZ-F. TIETZE

*Institut für Organische Chemie der Universität, Tammannstraße 2, D-3400 Göttingen, Federal Republic of Germany*

(Received 3 June 1988; accepted 19 August 1988)

**Abstract.** (6*aRS*,8*RS*,10*aRS*)-4,6,6*a*,7,8,9,10,10*a*-Octahydro-2,4,6,6,8-pentamethyl-1*H*-[2]benzopyrano-[3,4-*d*]pyrimidine-1,3(2*H*)-dione,  $C_{16}H_{24}N_2O_3$ ,  $M_r = 292.38$ , triclinic,  $P\bar{1}$ ,  $a = 7.759$  (1),  $b = 8.313$  (2),  $c = 12.985$  (2)  $\text{\AA}$ ,  $\alpha = 90.08$  (2),  $\beta = 98.38$  (2),  $\gamma = 113.24$  (2) $^\circ$ ,  $V = 759.8 \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 1.274 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$ ,  $\mu = 0.08 \text{ mm}^{-1}$ ,  $F(000) = 316$ ,  $T = 298 \text{ K}$ ,  $R = 0.050$  for 2229 observed reflections. The structure was investigated to determine the relative configuration which could not be established unambiguously by NMR. The *trans*-fused cyclohexane ring adopts a chair conformation, whereas the dihydropyran ring takes a twisted half-chair form.

**Experimental.** Crystal size  $0.2 \times 0.4 \times 0.8 \text{ mm}$ . Stoe–Siemens four-circle diffractometer, monochromated  $\text{Mo } K\alpha$  radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3914 reflections measured,  $2\theta_{\max} = 50^\circ$ ,  $h - 9 \rightarrow 9$ ,  $k - 9 \rightarrow 9$ ,  $l 0 \rightarrow 15$ , three check reflections with no significant intensity change. 2668 unique reflections ( $R_{\text{int}} = 0.059$ ), of which 2229 with  $F > 4\sigma(F)$  were used for all calculations [*SHELXS86* (Sheldrick, 1985); *SHELX76* (Sheldrick, 1976)]. Cell constants refined from  $\pm 2\theta$  values of 36 reflections in the range  $20\text{--}25^\circ$ . Absorption correction was not necessary. Extinction correction was performed yielding a secondary-extinction coefficient  $x$  of  $0.013$  (3), where  $F_c^* = F_c (1 +$

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

	x	y	z	$U_{eq}^*$
C(4a)	7239 (3)	4096 (2)	3908 (1)	31 (1)
O(5)	6930 (2)	5261 (2)	3284 (1)	38 (1)
C(3)	7754 (3)	3595 (2)	5732 (1)	36 (1)
C(1)	7396 (3)	1363 (2)	4360 (1)	38 (1)
N(2)	7692 (2)	1995 (2)	5410 (1)	39 (1)
O(3)	8006 (2)	4075 (2)	6648 (1)	51 (1)
N(4)	7493 (2)	4637 (2)	4942 (1)	35 (1)
C(10a)	7181 (3)	2135 (2)	2443 (1)	32 (1)
C(7)	7110 (3)	3309 (2)	662 (1)	37 (1)
C(10b)	7296 (3)	2571 (2)	3586 (1)	32 (1)
C(8a)	8072 (4)	2020 (3)	-792 (2)	57 (1)
O(1)	7238 (2)	-132 (2)	4176 (1)	56 (1)
C(8)	8484 (3)	2546 (3)	370 (1)	40 (1)
C(6)	6255 (3)	4721 (2)	2164 (1)	35 (1)
C(2)	7889 (4)	833 (3)	6224 (2)	59 (1)
C(6a)	7439 (3)	3780 (2)	1835 (1)	32 (1)
C(9)	8400 (3)	1010 (3)	1028 (2)	45 (1)
C(10)	8662 (3)	1456 (3)	2202 (1)	41 (1)
C(4)	7508 (3)	6343 (3)	5249 (2)	43 (1)
C(6Y)	6646 (3)	6462 (3)	1684 (2)	50 (1)
C(6X)	4131 (3)	3608 (3)	2030 (2)	47 (1)

\* Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

$0.002x^2/\sin^2\theta)^{-0.25}$ . Structure solution by direct methods. Refinement on  $F$  to  $R = 0.050$ ,  $wR = 0.074$ ; all non-H atoms anisotropic, H atoms were included using a riding model [C-H 0.96 Å,  $U(H) = 0.08 \text{\AA}^2$ , except for methyl protons  $U(H) = 0.12 \text{\AA}^2$ ], 191 parameters,  $S = 2.49$ , weighting scheme  $w^{-1} = \sigma^2(F) + 0.0005F^2$  which led to a featureless analysis of variance in terms of  $\sin\theta$  and  $F_o$ , max.  $\Delta/\sigma = 0.001$ , max. and min. heights in final  $\Delta\rho$  map 0.30 and  $-0.16 \text{ e \AA}^{-3}$  respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The site occupation factors for the two components of the disordered methyl group C(4) were initially refined (with their sum fixed at 1) and then fixed; an idealized hexagon was employed for the H-atom positions with  $U(H) = 0.08 \text{\AA}^2$ .

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.\* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

**Related literature.** For the preparation of some related compounds *via* a tandem-Knoevenagel-hetero-Diels-Alder reaction see Brumby (1987), Tietze (1984) and Tietze, Stegelmeier, Harms & Brumby (1982).

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51342 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected bond lengths (Å) and bond angles ( $^\circ$ )

C(4a)-O(5)	1.334 (2)	C(4a)-N(4)	1.376 (2)
C(4a)-C(10b)	1.353 (3)	O(5)-C(6)	1.477 (2)
C(3)-N(2)	1.374 (3)	C(3)-O(3)	1.216 (2)
C(3)-N(4)	1.388 (2)	C(1)-N(2)	1.414 (2)
C(1)-C(10b)	1.436 (3)	C(1)-O(1)	1.220 (3)
N(2)-C(2)	1.465 (3)	N(4)-C(4)	1.467 (3)
C(10a)-C(10b)	1.509 (2)	C(10a)-C(6a)	1.538 (3)
C(10a)-C(10)	1.535 (3)	C(7)-C(8)	1.528 (4)
C(7)-C(6a)	1.531 (2)	C(8a)-C(8)	1.522 (3)
C(8)-C(9)	1.523 (3)	C(6)-C(6a)	1.523 (3)
C(6)-C(6Y)	1.512 (3)	C(6)-C(6X)	1.522 (3)
C(9)-C(10)	1.532 (3)		
O(5)-C(4a)-N(4)	111.1 (2)	O(5)-C(4a)-C(10b)	125.5 (2)
N(4)-C(4a)-C(10b)	123.4 (2)	C(4a)-O(5)-C(6)	117.7 (1)
N(2)-C(3)-O(3)	122.7 (2)	N(2)-C(3)-N(4)	115.6 (2)
O(3)-C(3)-N(4)	121.6 (2)	N(2)-C(1)-C(10b)	115.9 (2)
N(2)-C(1)-O(1)	118.9 (2)	C(10b)-C(1)-O(1)	125.2 (2)
C(3)-N(2)-C(1)	125.3 (2)	C(3)-N(2)-C(2)	117.1 (2)
C(1)-N(2)-C(2)	117.6 (2)	C(4a)-N(4)-C(3)	121.2 (2)
C(4a)-N(4)-C(4)	121.2 (2)	C(3)-N(4)-C(4)	117.5 (1)
C(10b)-C(10a)-C(6a)	109.4 (1)	C(10b)-C(10a)-C(10)	114.3 (2)
C(6a)-C(10a)-C(10)	108.0 (2)	C(8)-C(7)-C(6a)	112.0 (2)
C(4a)-C(10b)-C(1)	118.0 (2)	C(4a)-C(10b)-C(10a)	120.5 (2)
C(1)-C(10b)-C(10a)	121.4 (2)	C(7)-C(8)-C(8a)	110.8 (2)
C(7)-C(8)-C(9)	110.1 (2)	C(8a)-C(8)-C(9)	111.8 (2)
O(5)-C(6)-C(6a)	106.9 (2)	O(5)-C(6)-C(6Y)	102.1 (1)
C(6a)-C(6)-C(6Y)	113.4 (2)	O(5)-C(6)-C(6X)	107.7 (1)
C(6a)-C(6)-C(6X)	114.7 (2)	C(6Y)-C(6)-C(6X)	110.9 (2)
C(10a)-C(6a)-C(7)	109.9 (1)	C(10a)-C(6a)-C(6)	111.7 (2)
C(7)-C(6a)-C(6)	114.6 (2)	C(8)-C(9)-C(10)	113.2 (2)
C(10a)-C(10)-C(9)	110.4 (2)		

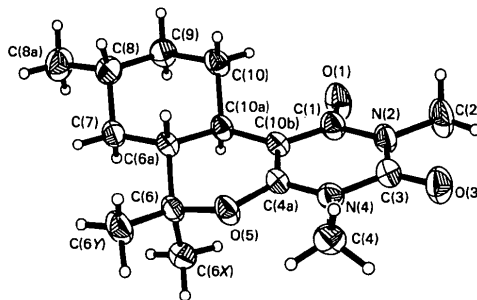


Fig. 1. A diagram of the title compound. Only one set of H-atom positions at C(4) is shown.

## References

- BRUMBY, TH. (1987). PhD Thesis, Univ. of Göttingen, Federal Republic of Germany.
- CLEGG, W. (1981). *Acta Cryst.* A37, 22-28.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination, modified by GMS. Univ. of Cambridge, England.
- SHELDRICK, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175-189. Oxford Univ. Press.
- TIETZE, L.-F. (1984). *Selectivity - a Goal for Synthetic Efficiency*, edited by W. BARTMANN & B. M. TROST, p. 299. Weinheim: Verlag Chemie.
- TIETZE, L.-F., STEGELMEIER, H., HARMS, K. & BRUMBY, TH. (1982). *Angew. Chem. Int. Ed. Engl.* 21, 863.